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REPORT ON DENTAL RESEARCH AT THE NATIONAL BUREAU OF STANDARDS

Progress Report

January 1 to June 30, 1961

The dental research program at the National Bureau of Standards is carried on in cooperation with the Council on Dental Research of the American Dental Association; the Army Dental Corps; the Dental Sciences Division of the School of Aviation Medicine, USAF; the Navy Dental Corps; and the Veterans Administration.

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U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

REPORT ON DENTAL RESEARCH
AT THE NATIONAL BUREAU OF STANDARDS

1. INTRODUCTION

Research on dental materials of various types, on dental equipment and on natural tooth structures continued at the National Bureau of Standards during the half year ending June 30, 1961.

Summaries of results obtained on work in progress, a list of reports issued and a list of papers published during the period are given below. Copies of the reports are appended,

2. REPORTS ISSUED

NBS Report 7142	The Synthesis of 2-Propoxy-5-Methylbenzoic Acid and its Reaction with Zinc Oxide.
NBS Report 7193	Properties of Dental Amalgam Made From Spherical Particles.
NBS Report 7329	Some Tensile Properties of Tooth Structure and Several Restorative Materials.
NBS Report 7334	Synthesis of Isomers of Eugenol.

3. PAPERS PUBLISHED

Second Revision of American Dental Association Specification No. 4 for Dental Inlay Casting Wax. J. W. Stanford, K. V. Weigel and G. C. Paffenbarger. J.A.D.A. 62:45 January 1961.

Surface Roughness of Gold Castings. J. J. Barone, R. L. Huff, and George Dickson. Dent. Prog. 1:78 January 1961.

Analysis of Pyrolyzates of Polystyrene and Poly(methyl methacrylate) by Gas Chromatography. F. A. Lehmann and G. M. Brauer. Anal. Chem. 33:673 May 1961.

Changes in Dentures During Storage in Water and in Service. J. B. Woelfel, G. C. Paffenbarger and W. T. Sweeney. J.A.D.A. 62:643 June 1961.

4. WORK IN PROGRESS

4.1 Human Tooth Enamel and Dentin

(a) Fluorescence Studies.

As considerable difficulty was experienced in the preparation of small pieces of selected areas of tooth sections suitable for nitrogen analysis by the Micro-Dumas method, serial

sections of dog femur were used as a preliminary test of the validity of the assumption that fluorescence intensity is related to the nitrogenous organic content. The data showed that in bone the fluorescence intensity decreased almost linearly with decrease in organic content. An unexplained increase in fluorescence intensity was observed during the first hour of extraction. This unexpected finding will be investigated further. Diamond impregnated cylinders are being prepared in a new attempt to cut out discs from selected areas of tooth sections. .

Work on the isolation and identification of the fluorescent components of tooth structure was resumed. As previous work indicated that the fluorescent material was attached to a high molecular weight peptide, it was decided to degrade the insoluble dentin matrix residue (from EDTA decalcification) in a gradual, stepwise manner. Two dimensional starch-gel electrophoresis, at high voltages, and chromatographic procedures were employed to analyze the hydrolyzates. Those peptide units showing high fluorescence intensity were isolated and the amino acid sequences are being determined. Amino acid sequences of similar hydrolyzates of dentinal protein from caries resistant and caries susceptible individuals will be compared in order to determine if the protein structure varies, thus explaining possible genetic influences on dental caries experience.

(b) Crystallographic Studies.

Crystallographic studies of tooth structures have been inactivated temporarily as a result of departure of the crystallographer conducting this work.

(d) Dental Calculus Studies.

Study of the organic content of dental calculus was continued. Results to date indicate that the supragingival and subgingival calculi are similar in composition. Also, no significant difference has been observed between calculi which form on natural teeth and that which forms on artificial dentures. Photographic studies of sections of teeth bearing calculus deposits by visible irradiation and by ultraviolet irradiation have revealed a film of material at the tooth and calculus junction. Further studies should be conducted regarding this film as it may be connected with the mechanism of attachment of calculus.

4.2 METALS

(a) Amalgam.

A detailed report on a study of the properties of dental amalgam made from spherical particles is appended.

(b) Gold Analysis.

The investigation of analysis of gold alloys by fire assay has produced improved manipulative procedures but has not so far produced sufficient improvement in precision of results. However, data obtained from x-ray fluorescence methods of analysis indicate that development of these rapid methods for replacement of the much slower wet analysis of gold alloys may be possible. Further investigations are being continued.

4.3 RESINS

(a) Denture Base Resins.

Measurements of the molar-to-molar and flange-to-flange distances were continued on 60 clinical and about 25 technic dentures. All of the clinical dentures have now been in service between three and four years. None of the dentures changed in dimension appreciably after the third month of use with the exception of those made of epoxy resin which continue to expand.

(b) Silica-Resin Direct Filling Material.

Synthesis of Surface Active Compounds and Monomers.

An attempt was made to synthesize a number of compounds containing polymerizable as well as chelating groups which may modify or react with the mineral or organic components of dentin. It is hoped that this study will give a better understanding of how to modify the tooth surface.

The reactions of 12-hydroxystearic acid and methacrylyl chloride and methacrylic anhydride have been studied to obtain the methacrylate ester of 12-hydroxystearic acid. This monomer has been obtained in 56% yield, but does not improve adhesion to dentin.

The possibility of obtaining polymerizable monomers containing chelating groups by reacting glycidyl methacrylate with glycine, N-phenylglycine and iminodiacetic acid has been explored. The reactions appear to be more complex than anticipated and only the monomer from N-phenylglycine and glycidyl methacrylate has been

isolated. This compound seems to improve adhesion to dentin.

An improved method was found for the preparation of monomers of the class: 1-O-methacryloyl-3-O-(R)-glycerol (where R is a phenyl derivative). Infrared spectroscopy was used to follow the addition reaction between the phenols and glycidyl methacrylate utilizing mainly the increasing absorption of the aromatic ether grouping at a frequency of approximately 1040 cm^{-1} . The utilization of infrared confirmed the structural formulas and also allowed the use of excess phenol in place of excess GMA (glycidyl methacrylate). The excess phenol could more easily be removed by subsequent washing with dilute aqueous alkali (than could the excess GMA be removed). The improvement in purity of the resulting monomeric product resulted in a significant reduction in toxicity as measured on rats. Monomers were prepared using GMA with phenol, p-tertiary butylphenol and bisphenol A.

Clinical Studies

Clinical trial of a silica-resin direct filling material has been started using Sevriton monomer and catalyst with the surface-treated silica powder (A187-161 plus pigments). Several of these fillings have been placed adjacent to either silicate cements or commercial direct filling resins. They will be observed and their condition recorded clinically, photographically and with stone casts every six months for a number of years. As soon as the monomers indicated above have been proven safe by adequate biological assay, they will be substituted for the methyl methacrylate monomer in the clinical trials.

4.4 Study of the Setting Reaction of Alkoxybenzoic Acids and Zinc Oxide

The synthesis and evaluation of cements containing 2-propoxy-5-methylbenzoic acid was completed. A progress report on this work is appended.

The possibility of reacting a solution of a solid chelating agent such as 2,3-dimethoxybenzoic acid or o-methoxybenzoic acid with zinc oxide has been investigated. However, these solids are only slightly soluble in water, alcohol, eugenol or o-ethoxybenzoic acid and no cementitious materials are obtained. Use of solvent systems does not look promising.

To reduce solubility of o-ethoxybenzoic acid containing cements a small amount of heavy metal oxide or acetate has been incorporated in the mixtures. Incorporation of lead oxide or lead acetate increases the powder-liquid ratio and produces mixes that harden completely within 6 minutes. The water solubility and disintegration of the mixes is decreased to a minimum of about 0.7% and crushing strength values do not change appreciably on addition of lead salts.

The separation of eugenol-chavibetol mixtures has been accomplished by gas chromatography with a diisodecyl phthalate column heated to 175°C using a hydrogen flame detector. Larger quantities of the isomers were separated using a Beckman chromatograph designed for synthetic work. Correlation of the physical properties of eugenol isomers and chemical structure is being investigated.

4.5 Investment

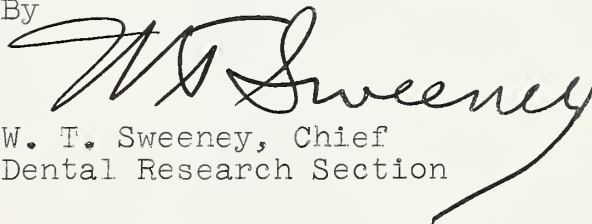
Study of the properties of casting investments of both the gold alloy type and the chromium-cobalt alloy type was continued. For both types of material, data are being obtained on the effect on thermal expansion of such factors as liquid-powder ratio, heating rate and preheating treatments. Of particular interest is the relationship between thermal expansion and setting and hygroscopic expansion since it is a combination of these expansions which compensates for the casting shrinkage of the alloy.

4.6 Evaluation of Materials

Materials evaluated for the Federal dental services and the American Dental Association by specification or other test methods included acrylic teeth, amalgam, chromium-cobalt alloy, denture base resin, gold alloy, investment and zinc phosphate cement.

For the Director

By



W. T. Sweeney, Chief
Dental Research Section

